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54 **Flame-retardant synthetic resin composition.**

**EP 0 293 531 A2**

57 A flame-retardant synthetic resin composition comprising a thermoplastic resin as a base polymer and, incorporated therein, (A) an organic fiber cut into a length smaller than 10 mm and/or an organic powder having a particle size smaller than 10 mesh and (B) a polyphosphoric acid compound having a high water resistance, wherein the amount of the thermoplastic resin is 40 to 95% by weight, the total amount of the components (A) and (B) is 5 to 60% by weight and the component (A)/component (B) weight ratio is in the range of from 5/1 to 1/20.

## FLAME-RETARDANT SYNTHETIC RESIN COMPOSITION

The present invention relates to a flame-retardant synthetic resin composition. More particularly, the present invention relates to a halogen-free flame-retardant synthetic resin composition in which the moldability (especially the parting property from a mold) is improved, the water resistance is enhanced, and the smoke-generating property is reduced.

5 In general, synthetic resins have a light weight and are excellent in water resistance, chemical resistance, and electrical insulation characteristics and can be easily molded and processed. Therefore, synthetic resins are widely used as materials for electric appliances, materials for automobiles, construction materials, and the like. However, synthetic resins are generally defective in that they are easily burnt.

Accordingly, there have been proposed various methods for rendering synthetic resins flame-retardant. 10 The method of internal addition of a halogen compound such as decabromodiphenylether or a halogen compound and antimony trioxide to synthetic resins is most widely adopted (see Japanese Examined Patent Publication No. 50-5103). Furthermore, there can be mentioned a method of addition of a metal hydroxide (Japanese Unexamined Patent Publication No. 50-34643, No. 50-133247, and No. 51-128194), a method of addition of a metal hydroxide and a polyphosphoric acid compound in combination (Japanese 15 Unexamined Patent Publication No. 54-22450), and a method of addition of an inorganic compound (Plastic Compounding, 15, July - August, 1986). Moreover, there can be mentioned a method in which a polyhydric alcohol such as pentaerythritol is used in combination with a polyphosphoric acid compound, urea, melamine, or the like for rendering a synthetic resin flame-retardant by utilizing the heat-insulating action of an intumescent and carbonized layer at the combustion and inhibiting the conduction of heat to the 20 substrate (Japanese Examined Patent Publication No. 61-47875 and Japanese Unexamined Patent Publication No. 60-36542).

However, these conventional techniques involve various problems. For example, in the case where a halogen compound is used, the hydrogen halide or the like generated by thermal decomposition corrodes the mold or the like at the molding or processing step, or, when a fire breaks out, evacuation is inhibited by 25 generation of noxious odors or poisonous gas. Furthermore, it has recently been reported that there is a risk of formation of dioxine as a thermal decomposition product of halogen compounds [Environ. Sci. Technol., 20(4), 404 - 408, (1986)]. Especially in West Germany, there is observed a trend toward the control of use of halogen compounds. In the case where an inorganic compound such as a polyphosphoric acid compound or a metal hydroxide is used, the flame-retardant effect is low and the inorganic compound 30 should be added in a large amount. Therefore, the specific gravity of the synthetic resin composition is increased and physical properties such as the strength and impact resistance are degraded.

Intumescent type flame retardants comprising polyphosphoric acid compound, polyhydric alcohols and the like, which have been extensively studied and practically marketed, are defective in that since most of these compounds have a high hygroscopicity and polyhydric alcohols are generally water-soluble, the resin 35 compositions comprising these compounds fail to have the high water resistance required for synthetic resins, the parting property from a mold is poor, and the molding efficiency is drastically reduced.

Various trials have been made to overcome these defects, but no satisfactory results have been obtained.

It is a primary object of the present invention to overcome the above-mentioned defects of the 40 conventional techniques and provide a flame-retardant synthetic resin composition in which generation of noxious or corrosive gas such as a hydrogen halide at the molding or processing step or the incineration step is prevented by rendering the composition intumescent and halogen-free, the parting property from a mold is improved, a practically sufficient water resistance is manifested, an excellent flame-retardant effect is attained by using the flame retardant in an amount much smaller than the amount used of the 45 conventional halogen compound or inorganic compound, and generation of smoke is drastically reduced.

We made research with a view to solving the above-mentioned problem, and, as a result, have now completed the present invention.

More specifically, in accordance with the present invention, there is provided a flame-retardant synthetic resin composition comprising a thermoplastic resin as the base polymer and, incorporated therein, (A) an 50 organic fiber cut into a length smaller than 10 mm and/or an organic powder having a particle size smaller than 10 mesh and (B) a polyphosphoric acid compound having a high water resistance, wherein the amount of the thermoplastic resin is 40 to 95% by weight, the total amount of the components (A) and (B) is 5 to 60% by weight and the component (A)/component (B) weight ratio is in the range of from 5/1 to 1/20.

The flame-retardant synthetic resin composition of the present invention is characterized in that no halogen compound with problems in toxicity and regulation are used, the water resistance and mold parting

property, which are insufficient in the conventional halogen-free flame-retardant synthetic resin compositions, are improved, and generation of gases or smoke at the molding step or at the time of combustion is drastically reduced. Namely, the present invention provides a so-called intumescent type flame-retardant synthetic resin composition in which, at the time of combustion, the burnt portion of the synthetic resin is  
5 intumescent and carbonized to form a porous carbonized layer acting as a heat-insulating layer, conduction of heat to the substrate is inhibited by this porous carbonized layer to prevent combustion, and generation of smoke is therefore prevented.

In the conventional intumescent flame-retardant synthetic resin compositions, polyhydric alcohols and derivatives thereof are mainly used as the intumescent and carbonizing source. According to the present  
10 invention, by using an organic fiber or organic powder instead of such a polyhydric alcohol or a derivative thereof, the water resistance, which is insufficient in the conventional intumescent flame-retardant synthetic resin compositions, is drastically improved. Furthermore, a polyphosphoric acid compound having a high water resistance is used as the carbonizing and intumescent catalyst in the present invention.

If the total amount of the components (A) and (B) is not larger than 5% by weight, the flame-retardant  
15 effect is low, and if the total amount of the components (A) and (B) exceeds 60% by weight, the physical properties and processability of the composition are drastically degraded, and the inherent characteristics of the synthetic resin composition are not sufficiently exhibited. If the (A)/(B) weight ratio is outside the range of from 5/1 to 1/20, the balance of the flame retardancy is not good. It is preferred that the total amount of the components (A) and (B) be 10 to 50% by weight, especially 15 to 40% by weight.

Thermoplastic resins customarily used can be used as the base polymer in the present invention. For  
20 example, there can be mentioned polyolefin resins such as polyethylene and polypropylene, polyester resins such as polyethylene terephthalate and polybutylene terephthalate, polyamide resins such as nylon-6, nylon-12 and aromatic polyamides, and polyphenylene oxides, modified polyphenylene oxides (Noryl), acrylonitrile-butadiene-styrene copolymers, polystyrene, polycarbonates, polymethyl methacrylate and poly-  
25 vinyl chloride.

Nylons, polyesters, acrylic resins, acetates, celluloses, other synthetic materials and natural materials that can be fibered or powdered can be used for the organic fiber or organic powder to be used in the present invention. The monofilament denier of the organic fiber is preferably smaller than 15 and especially  
30 preferably smaller than 10. The size of the organic powder is preferably smaller than 20 mesh and especially preferably smaller than 50 mesh. It is indispensable that the organic fiber or organic powder should not completely be molten at the molding step but should be softened and molten at the time of combustion. Accordingly, an organic fiber or organic powder having a softening point higher than the softening point of the base polymer and being capable of being softened and molten at a temperature lower than the combustion temperature is used.

When the organic fiber is added to a synthetic resin as the base polymer, the adaptability to the  
35 molding operation and the dispersibility in the base polymer should be taken into consideration, and if the cut length of the organic fiber exceeds 10 mm, the dispersion in the base polymer is insufficient and bad influences are imposed on the flame retardancy and physical properties. Accordingly, the cut length of the organic fiber should be smaller than 10 mm, preferably smaller than 7.5 mm, especially preferably smaller  
40 than 5 mm. In order to further improve the operation adaptability and the dispersibility, it is preferred that a bundle formed by bundling an organic fiber having a monofilament denier smaller than 20, especially smaller than 15, with an adhesive polymer be used. A water-dispersible, water-soluble or solvent-soluble polymer having a good compatibility with the base polymer is preferably used as the adhesive polymer. For  
45 example, there can be mentioned thermoplastic resins such as ethylene-vinyl acetate, polyvinyl acetate, polyamides, ethylene/vinyl chloride copolymers, ethylene/vinyl acetate/vinyl chloride copolymers, ethylene/ethyl acrylate copolymers, polyesters, and atactic polypropylene.

Polyphosphoric acid compounds having a high water resistance, such as polyphosphoric amides, ammonium polyphosphates, melamine-modified ammonium polyphosphates, and polyphosphoric car-  
bamates are preferably used as the polyphosphoric acid compound in the present invention.

50 Additives such as a lubricant, a plasticizer, an antioxidant, and an ultraviolet absorbent can be added to the synthetic resin composition of the present invention according to need.

The synthetic resin composition of the present invention is formed into a molded article having an  
arbitrary shape according to an ordinary molding method, for example, an injection molding method, an extrusion molding method, or a compression molding method. The synthetic resin composition of the  
55 present invention can be used for the production of parts of electric appliances, auto parts, interior articles, construction materials, clothing articles, miscellaneous articles, and the like.

According to the present invention, a flame-retardant synthetic resin composition which is excellent in moldability and processability, especially the parting property, and water resistance, and in which genera-

tion of smoke is reduced and generation of noxious odors smell or poisonous gas is prevented, is provided.  
The present invention will now be described in detail with reference to the following examples.

5 Example 1

A mixture comprising 70% by weight of polypropylene (J815HK supplied by Ube Industries Ltd.), 20% by weight of a high condensate of amido-phosphoric acid (Taien-S supplied by Taihei Kagaku Sangyo Kabushiki Kaisha) and 10% by weight of a nylon-6, fiber cut into 1 mm (monofilament denier = 2.9, supplied by Unitika) was kneaded at 180 °C for 15 minutes by a kneader (Model PBV-03 supplied by Irie Shokai Kabushiki Kaisha). The kneaded mixture was pressed at 200 °C for 3 minutes under 100 kg/cm<sup>2</sup> to form a plate having a thickness of 3 mm.

The obtained plate was subjected to a combustion test, a water resistance test, and a smoke-generating test.

15 Example 2

A mixture comprising 70% by weight of polypropylene as used in Example 1, 22% by weight of a high condensate of amido-phosphoric acid, and 8% by weight of a nylon-6 powder (the content of particles having a size of 80 to 100 mesh was at least 80%; supplied by Ube Industries) was kneaded at 180 °C for 15 minutes by using a kneader. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The obtained plate was subjected to a combustion test and a water resistance test.

25 Example 3

A mixture comprising 66% by weight of polypropylene as used in Example 1, 27% by weight of polyphosphoric amide (Sumisafe PM supplied by Sumitomo Chemical Co., Ltd.) and 7% by weight of a polyester fiber having a monofilament denier of 2 and cut into 3 mm length (supplied by Unitika) was kneaded at 180 °C for 15 minutes by a kneader and the kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test and a water resistance test.

35 Example 4

A mixture comprising 62% by weight of low-density polyethylene (M420 supplied by Mitsubishi Chemical Industries Ltd.), 28% by weight of a high condensate of amido-phosphoric acid, and 10% by weight of a nylon-6 fiber having a cut length of 1 mm (monofilament denier = 4.2; supplied by Unitika) was kneaded at 170 °C for 15 minutes by a kneader. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test and a water resistance test.

45 Example 5

A mixture comprising 62% by weight of an ABS resin (Kralastic K-3272M supplied by Sumitomo Naugatuck Kabushiki Kaisha), 30% by weight of a high condensate of amido-phosphoric acid, and 8% by weight of a nylon-6 fiber cut into a length of 3 mm (monofilament denier = 4.2) was kneaded at 200 °C for 15 minutes by a kneader. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test and a water resistance test.

Example 6

A mixture comprising 64% by weight of polymethyl methacrylate (Acrypet MD supplied by Mitsubishi Rayon Co., Ltd.), 28% by weight of polyphosphoric amide, and 8% by weight of a nylon-6 fiber cut into a length of 3 mm (monofilament denier = 4.2) was kneaded at 190 °C for 15 minutes. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test and a water resistance test.

10 Example 7

A mixture comprising 68% by weight of polypropylene as used in Example 1, 22.5% by weight of a high condensate of amido-phosphoric acid, and 9.5% by weight of a nylon-6 fiber bundled with 20% by weight, based on the weight of the bundles, of an ethylene/vinyl acetate copolymer (Evaflex P-2805 supplied by Mitsui-Du Pont Polychemical Kabushiki Kaisha) and cut into a length of 3 mm was kneaded at 180 °C for 13 minutes. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test and a water resistance test.

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Example 8

A mixture comprising 80% by weight of a rigid polyvinyl chloride compound (supplied by Riken Vinyl Kogyo Kabushiki Kaisha), 12% by weight of a high condensate of amido-phosphoric acid, and 8% by weight of a nylon-6 fiber cut into a length of 1 mm (monofilament denier = 1.5) was kneaded at 180 °C for 15 minutes by a kneader. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test, a water resistance test and a smoke-generating test.

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Comparative Example 1

A mixture comprising 70% by weight of polypropylene as used in Example 1, 24% by weight of decabromodiphenylether (Nonnen DP-10F supplied by Marubishi Oil Chemical Co., Ltd.), and 6% by weight of antimony trioxide (supplied by Mikuni Seiren Kabushiki Kaisha) was kneaded at 180 °C for 15 minutes by a kneader. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test, a water resistance test, and a smoke-generating test.

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Comparative Example 2

A mixture comprising 60% by weight of polypropylene as used in Example 1, 32% by weight of decabromodiphenylether, and 8% by weight of antimony trioxide was kneaded at 180 °C for 15 minutes by a kneader. The kneaded mixture was pressed at 200 °C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test, a water resistance test, and a smoke-generating test.

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Comparative Example 3

A mixture comprising 70% by weight of polypropylene as used in Example 1, 22.5% by weight of a high condensat of amido-phosphoric acid, and 7.5% by weight of pentaerythritol (supplied by Mitsui Toatsu Chemicals, Inc.) was kneaded at 180°C for 15 minutes by a kneader. The kneaded mixture was pressed at 200°C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test and a water resistance test.

Comparative Example 4

A mixture comprising 70% by weight of polypropylene as used in Example 1, 22.5% by weight of an ammonium salt of lowly condensed polyphosphoric acid (Taien-A supplied by Taihei Kagaku Sangyo Kabushiki Kaisha), and 7.5% by weight of pentaerythritol was kneaded at 180°C for 15 minutes by a kneader. The kneaded mixture was pressed at 200°C for 3 minutes to form a plate having a thickness of 3 mm.

The so-obtained plate was subjected to a combustion test and a water resistance test.

Test Results

## 1. Results of Combustion Test and Parting Property

The results of the combustion test according to the UL-94 test method and the results of the test of the parting property from a mold are shown in Table 1.

**Table 1**

	Total amount of additives (%)	UL-94	Parting property
Comparative Example 1	30	fail	good
Comparative Example 2	40	V-0	good
Example 1	30	V-0	good
Example 2	30	V-0	good
Example 3	34	V-0	good
Example 4	38	V-0	fair
Example 5	38	V-0	good
Example 6	36	V-0	good
Example 7	32	V-0	good
Example 8	20	V-0	good
Comparative Example 3	30	V-0	no parting property
Comparative Example 4	30	V-0	no parting property
Referential Example 1*	60**	V-0	-

\*: Plastic Compounding, 15 (July - August 1986)

\*\* :  $\text{Mg}(\text{OH})_2$

## 2. Results of Water Resistance Test

A sample was boiled for 60 minutes in boiling water and dried at 80° C for 30 minutes, and the weight decrease of the sample was measured. The obtained results are shown in Table 2. Incidentally, even if boiling was conducted for more than 60 minutes, no further change of the weight was observed.

Five samples having a size of 150 mm x 13 mm x 3 mm were cut from a plate having a size of 150 mm x 150 mm x 3 mm by a cutter, and the five samples were simultaneously tested. Although the cut surfaces were inferior to other surfaces in smoothness because of cutting by the cutter, these samples were tested without further treatment.



Table 2

	UL-94		Weight decrease (%)	Appearance after boiling	
	before boiling	after boiling			
5					
10	polypropylene	fail	fail	0.0	not changed
15	Comparative Example 1	fail	fail	0.0	slight color change on cut surface
20	Comparative Example 2	V-0	V-0	0.0	
25	Example 1	V-0	V-0	0.3	slight color change on cut surface
30	Example 2	V-0	V-0	0.1	
35	Example 3	V-0	V-0	0.2	
40	Example 4	V-0	V-0	0.2	
45	Example 5	V-0	V-0	0.1	
50	Example 6	V-0	V-0	0.0	
	Example 7	V-0	V-0	0.2	
	Example 8	V-0	V-0	0.3	
	Comparative Example 3	V-0	V-1	1.0	extreme color change in range of from cut surface to central part, falling of parts of edge, degradation of gloss in samples
	Comparative Example 4	V-0	V-1	4.0	

## 3. Results of Smoke-Generating Test

The surface test was carried out according to the method of Official Notice No. 1828 and 1231 of the Ministry of Construction (JIS A-1321-1975). A sample having a size of 220 mm x 220 mm x 3 mm and a weight of 550 to 580 g was used. The obtained results are shown in Table 3.

**Table 3**

	CA Value
<b>Polypropylene</b>	<b>132</b>
<b>Example 1</b>	<b>128</b>
<b>Polyvinyl chloride</b>	<b>above 240</b>
<b>Example 8</b>	<b>above 240</b>
<b>Comparative Example 1</b>	<b>above 240</b>
<b>Comparative Example 2</b>	<b>above 240</b>

**Claims**

1. A flame-retardant synthetic resin composition comprising a thermoplastic resin as a base polymer and, incorporated therein, (A) an organic fiber cut into a length smaller than 10 mm and/or an organic powder having a particle size smaller than 10 mesh and (B) a polyphosphoric acid compound having a high water resistance, wherein the amount of the thermoplastic resin is 40 to 95% by weight, the total amount of the components (A) and (B) is 5 to 60% by weight, and the component (A)/component (B) weight ratio in the range of from 5/1 to 1/20.

2. A composition as claimed in claim 1, wherein the total amount of the components (A) and (B) is 10 to 50% by weight.

3. A composition as claimed in claim 2, wherein the total amount is 15 to 40% by weight.

4. A composition as claimed in any one of the preceding claims wherein the base polymer is selected from polyolefin resins such as polyethylene and polypropylene, polyester resins such as polyethylene terephthalate and polybutylene terephthalate, polyamide resins such as nylon-6, nylon-12 and aromatic polyamides, and polyphenylene oxides, modified polyphenylene oxides, acrylonitrile-butadiene-styrene copolymers, polystyrene, polycarbonates, polymethyl methacrylate and polyvinyl chloride.

5. A composition as claimed in any one of the preceding claims, wherein the organic fiber or powder is composed of a nylon, polyester, acrylic resin, acetate, cellulose, or another synthetic or natural material capable of being converted into a fiber or powder.

6. A composition as claimed in any one of the preceding claims, wherein the monofilament denier of the organic fiber is smaller than 15.

7. A composition as claimed in claim 6, wherein the monofilament denier is smaller than 10.

8. A composition as claimed in any one of the preceding claims, wherein the particle size of the organic powder is smaller than 20 mesh.

9. A composition as claimed in claim 8, where the particle size is smaller than 50 mesh.

10. A composition as claimed in any one of the preceding claims, wherein the organic fiber has a length of smaller than 7.5 mm.

11. A composition as claimed in claim 10, wherein the length is smaller than 5 mm.

12. A composition as claimed in any one of the preceding claims, wherein the organic fiber or powder has a softening point higher than the softening point of the base polymer and is softened and melted before carbonization at the combustion.

13. A composition as claimed in any one of the preceding claims, wherein the organic fiber is one that has been bundled by a gathering polymer and cut into a length smaller than 10 mm.

14. A composition as claimed in claim 13, wherein the monofilament denier of the organic fiber is smaller than 20.

15. A composition as claimed in claim 14, wherein the monofilament denier is smaller than 15.

16. A composition as claimed in any one of claims 13 to 15, wherein the adhesive polymer is selected from ethylene-vinyl acetate copolymers, polyvinyl acetate, polyamides, ethylene-vinyl chloride copolymers, ethylene-vinyl acetate-vinyl chloride copolymers, ethylene-ethyl acetate copolymers, polyesters, and atactic polypropylene.

17. A composition as claimed in any one of the preceding claims, wherein the polyphosphoric acid compound is selected from polyphosphoric amides, ammonium polyphosphates, melamine-modified ammonium polyphosphates, and polyphosphoric carbamates.

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54 Flame-retardant synthetic resin composition.

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57 A flame-retardant synthetic resin composition comprising a thermoplastic resin as a base polymer and, incorporated therein, (A) an organic fiber cut into a length smaller than 10 mm and/or an organic powder having a particle size smaller than 10 mesh and (B) a polyphosphoric acid compound having a high water resistance, wherein the amount of the thermoplastic resin is 40 to 95% by weight, the total amount of the components (A) and (B) is 5 to 60% by weight and the component (A)/component (B) weight ratio is in the range of from 5/1 to 1/20.



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# EUROPEAN SEARCH REPORT

Application Number

EP 87 30 8960

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
X	FR-A-2 352 857 (MONTEDISON S.p.A.) * Claims; examples 5,6 * ---	1-17	C 08 K 3/32 C 08 L 101/00 C 09 K 21/14
X	EP-A-0 037 706 (MONTEDISON S.p.A.) * Pages 12-15 * ---	1-5	
X	DE-A-2 117 999 (MONSANTO CO.) * Claims; page 14, lines 1-15 * ---	1	
X	FR-A-2 091 666 (MONSANTO CO.) * Claims; example 4 * ---	1	
A	DE-A-3 603 493 (KATAFLOX PATENT-VERWALTUNGSGESELLSCHAFT mbH) -----		
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			C 08 K C 08 L
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 12-12-1989	Examiner DE LOS ARCOS Y VELAZQUEZ
<b>CATEGORY OF CITED DOCUMENTS</b> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ..... & : member of the same patent family, corresponding document			